

Table 1. Major Events in Run SA-0376 with 100 Fe/3 Cu/4 K/16 SiO₂ Catalyst (batch 4)

| TOS (h) | Event |
|---------|------------------------------------------------------------------------------------------------------------------------------------|
| | Slurry loading: 306 g of Durasyn 164 oil, 18.5 g of catalyst (particle size < 270 mesh) |
| | Catalyst pretreatment: H ₂ , 280°C, 0.78 MPa for 8 h |
| | Slurry sample withdrawal: 13 g slurry, 0.75 g catalyst |
| | Wax withdrawal through filter: 13 g of wax |
| 0 | Initiate synthesis gas flow, achieve process conditions: T = 260°C, P = 1.48 MPa, SV = 1.4 NI/g-cat/h, (H ₂ /CO) = 0.67 |
| 134 | Slurry sample withdrawal: 14.2 g slurry, 0.75 g catalyst |
| 159 | Change space velocity to SV = 2.3 NI/g-cat/h |
| 230 | Slurry sample withdrawal: 12.7 g slurry, 0.65 g catalyst |
| 254 | Change process conditions: P = 2.17 MPa, SV = 2.05 NI/g-cat/h |
| 350 | Slurry sample withdrawal: 30 g slurry, 1.0 g catalyst |
| | End of run: 264 g slurry recovered from the reactor |
| | Wax and catalyst removed during the run: 593 g wax, 3.2 g catalyst |

Table 2. Major Events in Run SB-0676 with 100 Fe/3 Cu/4 K/16 SiO₂ Catalyst (batch 4)

| TOS (h) | Event |
|---------|------------------------------------------------------------------------------------------------------------------------------------|
| | Slurry loading: 304 g of Durasyn 164 oil, 15.8 g of catalyst (particle size < 270 mesh) |
| | Catalyst pretreatment: H ₂ , 240°C, 0.78 MPa for 2 h |
| | Slurry sample withdrawal: 13 g slurry, 0.65 g catalyst |
| | Wax withdrawal through filter: 5.2 g of wax |
| 0 | Initiate synthesis gas flow, achieve process conditions: T = 260°C, P = 1.48 MPa, SV = 1.4 NI/g-cat/h, (H ₂ /CO) = 0.67 |
| 120 | Slurry sample withdrawal: 11 g slurry, 0.6 g catalyst |
| 166 | Change process conditions: P = 2.17 MPa, SV = 1.8 NI/g-cat/h |
| 221 | Slurry sample withdrawal: 26 g slurry, 1.2 g catalyst |
| 305 | Slurry sample withdrawal: 28.4 g slurry, 1.0 g catalyst |
| 305 | End of run: 438 g slurry recovered from the reactor |
| | Wax and catalyst removed during the run: 78 g wax, 3.45 g catalyst |

Table 3. Effect of Calcination Conditions on the Textural Properties of Catalysts C (100 Fe/3 Cu/4 K/16 SiO₂) and B (100 Fe/5 Cu/6 K/24 SiO₂).

| Calcination Conditions | BET Surface Area (m ² /g) | | Pore Volume Cm ³ /g |
|--------------------------------------------------------------|--------------------------------------|----------|-----------------------------------|
| | Single Point | BET Plot | |
| 100 Fe/3 Cu/4 K/16 SiO₂, batch 4 (S3416-4) | | | |
| 300 °C for 5 hours | 310 | 306 | 0.45 |
| 400 °C for 5 hours | 263 | 268 | 0.42 |
| 500 °C for 5 hours | 183 | 188 | 0.36 |
| 500 °C for 30 minutes | 233 | | |
| 500 °C for 60 minutes | 231 | 216 | 0.40 |
| 700 °C for 30 minutes | 118 | | |
| 700 °C for 60 minutes | 108 | 106 | 0.30 |
| 100 Fe/5 Cu/6 K/24 SiO₂, batch 3 (S5624-3) | | | |
| 300 °C for 5 hours | 258 | 284 | 0.51 |
| 400 °C for 5 hours | 271 | 253 | 0.48 |
| 500 °C for 5 hours | 219 | 193 | 0.43 |
| 500 °C for 60 minutes | 227 | 212 | 0.46 |
| 700 °C for 30 minutes | 115 | 108 | 0.33 |
| 700 °C for 60 minutes | 100 | 96 | 0.33 |

Table 4. Summary of Results from TPR Scans *

| Sample | Peak Position (°C) | | Degree of Reduction (%) | |
|-------------------------------------|-----------------------------|------------------------------|------------------------------|---------------------------------------|
| | First Stage Reduction Peaks | Second Stage Reduction Peaks | Based on First Stage Peak(s) | Based on First and Second Stage Peaks |
| 100 Fe | 307, 360 | 595 | 3.0, 12.0 | 61.7 |
| 100Fe/0.2K | 380 | 610 | 11.6 | 86.1 |
| 100Fe/0.5K | 384 | 620 | 10.4 | 90.8 |
| 100Fe/1K | 384 | 632 | 10.5 | 90.4 |
| 100Fe/0.3Cu | 360 | 505, 610 | 15.2 | 48.7, 66.9 |
| 100Fe/3Cu | 260 | 560 | 9.4 | 95.0 |
| 100Fe/5Cu | 240, 280 | 565 | 7.5, 11.1 | 95.0 |
| 100Fe/3Cu/16SiO ₂ | 320 | 595 | 30.3 | 91.9 |
| 100Fe/5Cu/24SiO ₂ | 312 | 595 | 30.0 | 97.0 |
| 100Fe/3Cu/4K/16SiO ₂ | 320 | 580 | 23.8 | 74.1 |
| 100Fe/5Cu/6K/24SiO ₂ | 320 | 565 | 30.0 | 80.2 |
| 100Fe/3Cu/4K/6Ca/16SiO ₂ | 317, 350 | 610 | 4.9, 14.0 | 93.3 |
| 100Fe/5Cu/6K/6Ca/24SiO ₂ | 340, 370 | 630 | 5.9 | 85.7 |

* Experimental Conditions: Rate of Heating = 20°C/min.; Reducing Gas Composition = 5%H₂/95%N₂, Flow Rate = 40 ml/min.; Catalyst Weight = 10-20 mg;

Table 5. Effect of the Addition of Copper, Potassium and Silica on the Degree of Reduction

| Catalyst | Degree of Reduction | | |
|------------------------------------------|---------------------|----------------|------|
| | TGA Isothermal | TPR Isothermal | TPR |
| 100 Fe | - | - | 61.7 |
| 100 Fe/3.0 Cu | 86.5 | 19.4 | 95.0 |
| 100 Fe/3 Cu/16 SiO ₂ | 47.7 | 25.0 | 91.9 |
| 100 Fe/3 Cu/4 K/16 SiO ₂ | 48.1 | 22.1 | 74.1 |
| 100 Fe/3 Cu/4 K/6 Ca/16 SiO ₂ | 46.2 | - | 93.3 |
| 100 Fe/5.0 Cu | 88.5 | 20.9 | 95.0 |
| 100 Fe/5 Cu/24 SiO ₂ | 47.0 | 27.8 | 97.0 |
| 100 Fe/5 Cu/4 K/24 SiO ₂ | 47.3 | 21.1 | 80.2 |
| 100 Fe/5 Cu/6 K/6 Ca/24SiO ₂ | 34.5 | - | 85.7 |